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भारतीय मानक

पेट्रोलियम और उसके उत्पादकों की परीक्षण पद्धतियां

[पी : 55/वर्ग 1]

पेट्रोलियम उत्पादकों का साबुनीकरणी मान का निर्धारण

(पहला पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 55/Section 1]

DETERMINATION OF SAPONIFICATION VALUE OF PETROLEUM PRODUCTS

(First Revision)

ICS 75.080

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2004 **Price Group 2**

Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 55/Sec 1] (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Saponifiable value, saponifiable and unsaponifiable matter, *see* [P : 55] of IS 1448 was published in 1963 in order to align the method given in ASTM D 963 : 1954, which has since been withdrawn in 1963. It was decided to publish the separate test method, namely, Saponifiable value of petroleum products and saponifiable and unsaponifiable matter in oil fat and waxes. Considerable assistance has been derived from ASTM D 94 and IP 136 for formulation of this test method.

The composition of the Committee responsible for formulation of this standard is given in Annex A .

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS
[P : 55/Section 1]
DETERMINATION OF SAPONIFICATION VALUE OF PETROLEUM PRODUCTS
(First Revision)

1 SCOPE

This method [P : 55/Sec 1] is intended for the saponification value in straight mineral oils, compounded mineral oils and waxes.

2 SUMMARY OF METHOD

A known mass of sample is dissolved in 2-butanone (Methyl ethyl ketone) is heated with known amount of alcoholic potassium hydroxide. The excess alkali is titrated with standard acid and saponification number calculated. This method is not applicable in the presence of compounds of sulphur, phosphorous, chlorine, etc. which reacts with acid or alkali.

NOTE — If larger amounts of unsaponifiable or saponifiable matter are required for further examination, larger samples may be taken provided the quantities of the reagents are suitably adjusted.

3 TERMINOLOGY

3.1 Saponify — To hydrolyze a fat with alkali to form an alcohol and the salt of a fatty acid.

3.2 Saponification Number — The number of milligrams of potassium hydroxide consumed by one gram of a sample under the conditions of the test.

4 APPARATUS

4.1 Erlenmeyer Flask and Condenser

Erlenmeyer flask of 300 ml capacity. Flask which has become etched by long use should not be used. The straight type condenser shall be fitted to the flask with ground glass joint.

4.2 Hot-Plate or Water Bath

5 REAGENTS

5.1 Purity of Reagents

Analytical grade reagent chemicals shall be used in all tests.

5.1.1 *Alcohol* — 95 percent ethanol.

5.1.2 *Standard Potassium Hydroxide Solution, 0.5 N* – Prepare approximately 0.5 N alcoholic potassium hydroxide by dissolving 30 g of potassium hydroxide in 1 litre alcohol. Allow the solution to settle in dark place. Filter the solution and keep it for 24 h before use.

5.1.3 *Standard Hydrochloric Acid,* 0.5 N

Mix 45 ml of hydrochloric acid (Sp gr 1.19) in 1 litre of alcohol.

5.1.4 *Butanone-2 (Methyl Ethyl Ketone) Technical Grade*

5.1.5 *Petroleum Spirit,* 60/80.

5.1.6 *Phenolphthalein Indicator Solution*

6 SAMPLE SIZE

Use such a size of the sample that not more than half of the potassium hydroxide added in the reagent, reacts during the test.

The following quantities are suggested:

Saponification Number	*Sample Size,* g
181 to 400	1
111 to 180	2
71 to110	3
31 to 70	5
16 to 30	10
Mineral oils	20

7 PROCEDURE

7.1 Weigh the oil sample to the nearest 0.1g into the reaction flask. Add 25±1 ml methyl ethyl ketone, followed by 25 ± 0.3 ml alcoholic KOH solution measured accurately from the burette or pipette. Connect the condenser to the flask and heat for 30 min after refluxing begins. Immediately disconnect the condenser add 50 ml of petroleum sprit and immediately titrate the solution with standard hydrochloric acid using 1 ml of phenolphthalein indicator. When testing waxes it may be necessary to reheat during titration in order to prevent solidification of the sample.

7.2 Make a blank determination in duplicate at the same time following exactly the same procedure but omitting the sample.

8 CALCULATION

Calculate the saponification number as follows:

$$\text{Saponification number} = \frac{56.1(V - V_1)N}{M}$$

V = volume, in ml of acid used in titrating the blank;

V_1 = volume, in ml of acid used in titrating the sample;

N = normality of the hydrochloric acid; and

M = mass of the sample, in g.

9 REPORT

Report the result to the nearest 0.5 as the saponification value.

10 PRECISION

Follow the graph as per IP 136/ASTM D 94.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehra Dun	Shri Sudhir Singhal (***Chairman***) Dr A. Dutta (*Alternate*)
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(*Continued on page* 4)

(*Continued from page* 3)

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